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REPORT No. 9/R/62

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Lead Styphnate:

Part 4: The Monobasic Lead Salts of
Trinitroresorcinol, Polymorphic Modifications and
the Development of R.D. 1346 and R.D. 1349

G.W.C. Taylor
A.T. Thomas

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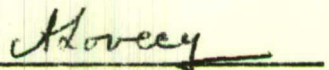
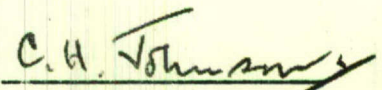
Lead Styphnate:

Part 4: The Monobasic Lead Salts of Trinitroresorcinol,
Polymorphic Modifications and the Development of R.D.1346 and R.D.1349

by

G.W.C. Taylor and A.T. Thomas

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Reference: WAC/128/019

1. SUMMARY

The preparation and properties of three polymorphic modifications of monobasic lead trinitroresorcinate have been studied. The common (alpha) polymorph, as prepared by ordinary methods, is of poor physical form and is very sensitive to ignition by electrostatic spark. By varying the method of preparation, and by the employment of small quantities of additives, a range of products of different physical form and properties, including reduced sensitivity to spark ignition, can be obtained. Two such products (designated R.D.1346 and R.D.1349, both the beta polymorph) have possible technical applications for electrical initiation and explosive priming and delay purposes. They have been developed to the manufacturing scale and the respective processes and properties of technical interest are described. Compared with normal lead trinitroresorcinate the new varieties offer improved chemical and thermal stability combined with promising explosive characteristics.

2. OBJECT OF INVESTIGATION

To study the methods of preparation and properties of the polymorphic modifications of monobasic lead trinitroresorcinate and the development of technically useful products.

3. INTRODUCTION

The preparation and confirmed identification of monobasic lead trinitroresorcinate is complicated by its occurrence in polymorphic modifications which are very dependent upon preparative conditions, including especially the purity of ingredients and the use of additives. These conditions also markedly affect the properties of technical interest, such as physical form and sensitiveness. Accordingly the investigation of reliable methods of preparation is a first objective towards assessing the feasibility of this explosive as a filling for technical stores.

Three basic lead trinitroresorcينات, generally termed basic lead styphnates, are described in the technical literature; the yellow acicular type by Böttger and Will (1), the red diamond-shaped form by Brün (2), and an amorphous yellow variety by Tauson (3).

These three forms were prepared and examined by Taylor and White (4), and from analysis, density measurement and X-ray diffraction examination, they established that these substances were trimorphic forms of basic lead styphnate with a composition corresponding to the empirical formula $C_6H_3N_3O_{10}Pb_2$. More detailed investigations have since been carried out.

Monobasic lead styphnate possesses potentially useful properties as an electrically-initiated composition, as a mixture ingredient for explosive trains, and as a fast delay composition. It has been used in America as an ingredient of primer mixtures but it is not a Service explosive in this country. The common yellow form has serious disadvantages for technical use, mainly its inherent bulky character, both in the moist and dry conditions, presenting manufacturing difficulties. Also, in the dry state, it is highly

/sensitive

sensitive to electrostatic ignition constituting an explosive hazard during handling. Hitchens and Garfield (5) have prepared a free-flowing crystalline form by precipitation in the presence of a crystallisation-controlling agent and claim to have overcome the drawback associated with the manufacture of the bulky yellow form.

The red crystalline form described by Brün is a free-flowing product but the preparation has been developed to the 30-gram scale only. Certain difficulties associated with scaling up, and the dependence of the method, and nature of the product, upon the purity of the parent styphnic acid are disadvantageous.

Chemically, the basic salt possesses certain advantages over the normal salt of lead styphnate in that it is more stable in an ammoniacal atmosphere, and also, due to its basic nature, it does not accelerate the hydrolytic decomposition of moist lead azide in juxtaposition. Another important advantage is its high thermal stability up to a temperature of 200°C without loss of hydrate or change in physical form.

The present report covers a more detailed study of the alpha, beta and gamma polymorphic modifications. Their methods of preparation on the laboratory and semi-technical scale have been examined, and conditions for crystal-habit control of both the alpha and beta modifications have been established. Two habit forms of the beta polymorph, one red rounded granular and the other oval or cigar-shaped crystals, were of sufficient promise, for dry and paste loading respectively, for their methods of preparation to be developed to full manufacturing scale. These compositions have been designated R.D.1346 and R.D.1349 respectively.

R.D.1346 has been tested as an ingredient for ammunition priming mixture; it serves a dual purpose, acting as a fuel deficient in oxygen, and it is also sufficiently sensitive to friction and percussion to be capable of use without the usual combustion initiator such as mercury fulminate or tetrazene. It has also been found to give satisfactory conducting composition when mixed with graphite. R.D.1349 is of a suitable physical form to give by itself a satisfactory dipping mixture for sensitive fuseheads.

Sensitiveness to impact, friction and electrostatic discharge have been determined and compared with normal lead styphnate R.D.1303 (6), and results are included in this report.

4. EXPERIMENTAL

4.1 Preparation of Alpha Polymorph of Monobasic Lead Styphnate

4.1.1 By Hydrolysis of Normal Lead Styphnate

Alpha monobasic lead styphnate is formed when either alpha (6) or beta (7) normal lead styphnate is subjected to the prolonged action of hot water. Thus 5 g. alpha normal salt (R.D.1303) digested with 100 ml. water and heated under reflux for two hours on a boiling-water-bath gave complete conversion to the alpha monobasic lead styphnate. The formation of the alpha monobasic salt is shown by the appearance of characteristic acicular crystals. The other polymorphic modifications of the monobasic salt have not been observed on hydrolysis of the normal salt, although long storage under water

/of

of the beta normal salt can give rise to the gamma normal salt which is in the form of dark yellow squat crystals.

4.1.2 By Interaction of Soluble Lead and Styphnic Acid Salts

The alpha monobasic salt is prepared by the interaction of two molecular proportions of a soluble lead salt with one molecular proportion of an alkali styphnate salt at a temperature of 90°C. The product consists of masses of orange yellow needles (Fig. 1) which are very bulky and difficult to separate from the mother-liquor. The product finally dries to a well-knit cake which is hazardous to handle because of the mechanical and electrostatic spark sensitiveness of this material.

Attempts were made to improve the physical form of the product by experimental precipitations using lead nitrate and sodium styphnate as reactants and varying parameters such as temperature, concentration of reactants and rate of addition. No improvement to the product could be obtained.

4.1.3 The Employment of Additives

Following a method described by Hitchens and Garfield (5), by precipitating the alpha basic lead styphnate in the presence of an added crystallisation-controlling agent (nitrosation/nitration product of resorcinol) a modified crystal habit in the form of tan-yellow rectangular parallelepiped crystals was obtained. This modified form has been prepared here on the laboratory (25 g.) and semi-technical scale (1¼ lb.) and the explosive characteristics evaluated. The controlling additive is prepared by consecutive nitrosation and nitration of resorcinol and is in the form of a dark brown powder of uncertain composition containing about 10.6 per cent nitrogen, 2.2 per cent hydrogen and 39.9 per cent carbon. The method of preparation on the semi-technical scale for modified alpha monobasic lead styphnate is as follows:

272.7 g. styphnic acid and 4.5 g. nitrosation/nitration product of resorcinol are dispersed in 3600 ml. water warmed to 62° - 65°C in a 10-litre stainless steel jacketed pan (12), and stirred. 119.7 ml. glacial acetic acid are added followed by 280 ml. of ammonium hydroxide solution (255 g. NH₃ per litre). The resulting pH should be between 5.5 and 6.5 at 25°C. 729 g. lead nitrate in 2700 ml. water are added in 34 minutes to the base solution maintained at 68° - 70°C. Three minutes after start of the lead nitrate solution, 1175 ml. ammonium hydroxide solution, containing 153 ml. of 255 g. NH₃ per litre, are run into the pan in 16 minutes. Agitation is continued keeping the temperature at 68° - 70°C for a period of 25 minutes after the end of lead nitrate addition. The product is allowed to settle, the supernatant liquid decanted and the product washed repeatedly with water and finally with methylated spirit and dried at 50°C. 1¼ lb. of minute tan-coloured rectangular parallelepiped crystals with a bulk density of 1.67 g/ml. were obtained (Batch XBL5 57), Fig. 2.

Experimental precipitations were carried out replacing the indefinite nitrosation/nitration product by 2-nitroresorcinol and dinitroresorcinol as the crystallisation agent but no modified rectangular crystals were obtained. Also no modification of crystal form occurred when impure dark brown styphnic acid was used without the added nitrosation/nitration product.

Further experiments to determine the effect of the presence of additives were carried out and it was found that, with precipitations using lead nitrate

/and

and sodium styphnate as reactants, the addition of varying amounts of 2-nitro-resorcinol or the nitrosation/nitration of resorcinol gave no improvement to the physical form of the product.

4.2 Preparation of Beta Polymorph

4.2.1 Brün's Method

The beta polymorph prepared by W. Brün (2) is in the form of red diamond-shaped crystals, and the method of preparation is as follows:

"A solution of 12.2 g. of styphnic acid and 8 g. of sodium hydroxide in 400 ml. of water at a temperature between 60°C and 70°C is dropped with stirring into a solution of 35 g. of lead nitrate in 350 ml. of water. At first the sodium styphnate sodium hydroxide solution must be added very slowly, drop-by-drop, to the lead nitrate solution. The precipitate appears to be formed first as an amorphous yellow basic lead styphnate. If a suitable time is permitted to elapse before more sodium styphnate/sodium hydroxide solution is added, the amorphous yellow precipitate suspended in the solution crystallises to small regular diamond-shaped crystals of a reddish brown colour which quickly settles out. As the operations proceeds the added solution may be run in more quickly".

Earlier work in E.R.D.E. (4) has indicated that the conditions of preparation of this modification (free from the alpha form) are most critical. In the present investigation laboratory preparations were carried out using pure styphnic acid and in five repeat experiments, varying the addition period of the sodium styphnate/sodium hydroxide solution from 90 to 240 minutes, no beta form was obtained, but only typical alpha acicular product. The method was not further pursued; but the simpler procedure of adding the sodium styphnate/sodium hydroxide solution at room temperature to the lead nitrate solution, which was stirred and maintained at 70°C, was adopted. However, only the yellow acicular alpha form resulted when pure styphnic acid was used as the parent compound.

4.2.2 Preparation of Beta Polymorph from Impure Styphnic Acid

When commercial impure dark brown styphnic acid was used for the preparation of the sodium styphnate/sodium hydroxide solution described by Brün and added to lead nitrate base solution at 70°C the red crystalline beta form was obtained. This type of styphnic acid, resulting from successive sulphonation and nitration, contains as impurities nitrosation and nitrosation/nitration products of resorcinol such as dinitrosoresorcinol, nitroresorufin and nitroresazurin. It appears therefore that the presence of some impurity is necessary for the formation of the beta form.

4.2.3 Employment of Additives

In order to obtain reproducible full-scale manufacturing results it is considered essential that pure styphnic acid, with addition of some crystallisation-controlling agent, should be used rather than impure styphnic acid, which would necessitate initial small-scale preparations to ascertain the suitability of each batch. Preferably the crystallisation-controlling agent should be readily available, and should be a pure, definite compound which can be chemically specified.

/Experiments

Experiments were carried out to determine the effect of additives on the physical form of the precipitate obtained by the addition of sodium styphnate/sodium hydroxide solution (prepared with pure styphnic acid) to lead nitrate solution at 70°C over a prolonged period of 180 minutes. It was found that 2-nitroresorcinol and the nitrosation/nitration product of resorcinol were effective to give the red beta polymorph, whereas 2,4-dinitroresorcinol, 4,6-dinitroresorcinol and 2-nitrosoresorcinol had no effect and the ordinary yellow alpha form resulted.

The analysis for the red beta form gave: lead 60.2 per cent, styphnate 34.3 per cent; theory for monobasic lead styphnate $[C_6HO_3N_3Pb \cdot Pb(OH)_2]$ lead 59.92 per cent, styphnate 35.16 per cent.

Monobasic lead styphnate is slightly soluble in aqueous ammonium acetate solution. The metathesis of aqueous solutions of ammonium styphnate and lead acetate produces ammonium and acetate ions in the mother liquor which should promote the growth of larger sized crystals of the basic salt. This method of preparation of the basic salt has been studied, and it was found that the added presence of 2-nitroresorcinol had the effect of modifying the alpha acicular basic salt to form cigar-shaped tan-coloured crystals of the beta habit. This product settled readily from the mother liquor, and, after washing, dried to give discrete crystals without caking. When the nitrosation/nitration product of resorcinol was added in place of 2-nitroresorcinol a modified alpha form similar in crystal shape to that formed by the Hitchens and Garfield method was obtained.

4.2.4 Development of R.D.1346

As 2-nitroresorcinol is readily available and is a more definite compound than the nitrosation/nitration product of resorcinol it was selected as the crystallisation-controlling agent in the development of plant-scale preparations of the red form of beta monobasic lead styphnate. Preparations were carried out on the semi-technical scale using a 10-litre capacity precipitating pan and information was obtained relating to the dependence of the reaction and nature of the final product on the type of parent styphnic acid used. With pure styphnic acid (to specification) (8), without additive, typical alpha acicular needles were obtained, whereas with crude dark brown styphnic acid rounded red granules of the beta form were obtained. Precipitations using different batches of "pure" styphnic acid (all within specification limits) as the parent compound, and 2-nitroresorcinol as the additive, gave beta granular products of varying bulk density. The presence of 0.77 per cent 2-nitroresorcinol (on styphnic acid content) gave with one batch of styphnic acid (9/44) a product with a bulk density of 1.94 g/ml., and with another batch (R.D.7) a bulk density of 0.80 g/ml. (Fig. 3). By increasing the additive to 1.54 per cent this latter batch gave a product of bulk density 1.70 g/ml. (Fig. 4). With another batch of styphnic acid (A.2) a yellow gel appeared during the precipitation and persisted to contaminate the beta granular end-product. This gel appeared to be a yellow-stained lead hydroxide precipitate which could be removed by the careful addition of dilute acetic acid. However, it was found that the production of this undesirable gel was avoided by using 100 per cent excess lead nitrate at a higher concentration, as the base solution. The sodium styphnate/sodium hydroxide solution darkens considerably on keeping, probably due to formation of chelate ortho-nitro-derivatives with sodium ion. In order to avoid this disadvantage, the use of sodium carbonate was examined for the preparation of the soluble disodium salt of styphnic acid for stock solution. A molar proportion of sodium carbonate was added to styphnic acid

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to give the aqueous soluble disodium styphnate, which remained stable without darkening in colour after standing two months at room temperature (20°C). The extra sodium hydroxide solution necessary for formation of the monobasic salt was added to the disodium styphnate solution immediately before beginning the metathesis reaction. A satisfactory process and product were obtained by this procedure. The preparation and process instructions of R.D.1346 on a semi-technical scale have been described (9) and are reproduced in the Appendix.

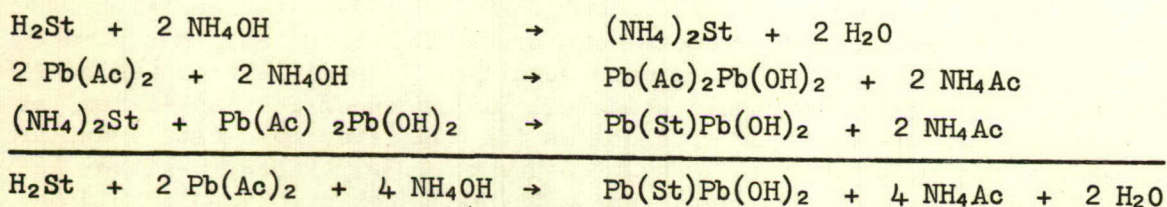
Preparations have been carried out on the standard 45-litre stainless steel initiator pan scaling up six times the semi-technical scale. 3½ lb. per batch can be manufactured. However, the variability of styphnic acid from batch to batch necessitates the determination of the optimum quantity of 2-nitroresorcinol for controlling the final product to a low, medium or high bulk density by carrying out a series of small precipitations. In general a low bulk density material is required for electrically conducting compositions, a medium bulk density material for priming mixtures and a high bulk density for fast delay compositions.

Details of the full manufacturing scale are given in the Appendix.

4.2.5 Development of R.D.1349

Characteristic cigar-shaped crystals of beta monobasic lead styphnate, produced by the addition of lead acetate solution to ammonium styphnate solution containing 2-nitroresorcinol as additive, have been designated R.D.1349 and the method of manufacture has been developed to a 4-lb. batch size.

For the formation of this basic salt by this method four moles of ammonia per mole of styphnic acid are required, which is two moles in excess of that required for forming ammonium styphnate. During laboratory experimentation it was found that one molecular proportion of aqueous ammonia could be added to one molecular proportion of lead acetate to give a clear solution which, for precipitating purposes, functioned as a solution of basic lead acetate $[\text{Pb}(\text{Ac})_2\text{Pb}(\text{OH})_2]$. The equations for the reaction of precipitation can be written:



Initial preparations on the laboratory scale (0.04 mole styphnic acid) gave the required cigar-shaped crystals by a precipitation method which readily could be scaled up. Precipitations on the semi-technical scale (10-litre capacity pan: 0.6 mole styphnic acid) were carried out, and conditions for the satisfactory preparation were determined. During the early stages of precipitation gel fragments were formed which were transformed into the cigar-shaped crystals before the end of the addition of the ammonia/lead acetate solution. In some precipitations traces of gel fragments persisted at the end of the addition, but could be removed by the addition of dilute acetic acid to the final supernatant liquid.

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In order to avoid the addition of acid, the effect of the amount and location of the ammonia distributed between the ammonia/ammonium styphnate base solution and the ammonia/lead acetate addition solution was investigated. The most favourable conditions for complete change of gel fragments to the desired cigar-shaped crystals obtained when all the ammonia was allocated to the ammonium styphnate base solution. However, concurrent measurements of the electrostatic sensitiveness of these products had clearly indicated that, where part of the ammonia was located in the lead acetate run-in solution, the products were markedly less sensitive than those obtained when all the ammonia was added to the base solution (see Table 5, p.14). A method of preparation was developed which gave a product free from gel contaminant and with the safest handling properties to spark hazard. Of the 2.4 moles of ammonia required per 0.6 mole styphnic acid, 2.15 moles of ammonia were added to the base solution and 0.25 mole to the lead acetate solution. The method of preparation on the semi-technical scale is given in the Appendix; this has already been reported (12) (see Fig. 5).

Four large-scale precipitations on the standard initiator manufacturing plant were carried out and 4½-lb. batches were prepared successfully. Details of the preparation are given in the Appendix.

4.3 Preparation of Gamma Polymorph

Tauson (3) prepared this amorphous form of monobasic lead styphnate by the addition of aqueous lead nitrate to a stirred ammoniacal solution of ammonium styphnate maintained at a temperature between 35° and 50°C.

A laboratory preparation (BLS 21) was carried out as follows:

To a solution of 4.9 g. (0.02 mole) styphnic acid digested with 96 ml. 2.16 per cent (0.12 mole) aqueous ammonia maintained at 40° - 45°C was added 50 ml. containing 13.5 g. (0.0405 mole) lead nitrate over a period of 30 minutes. 13.8 g. of a yellow amorphous product was obtained which gave a different X-ray diffraction pattern from the alpha and beta forms. A tenfold scale-up preparation (XBLS 49) was made on the semi-technical plant and a similar product was obtained (Fig. 6).

This amorphous form is bulky in nature and Tauson describes in his patent the procedure of washing the product into a funnel lined with filter paper, dehydrating by the use of alcohol, and then de-alcoholising by repeated washings with ether. It is dried at 140°F (60°C) for 16 to 18 hours and then mechanically ground before use.

In order to avoid this hazardous procedure attempts have been made here to modify the physical form of the product. The use of additives such as 2-nitroresorcinol, the nitrosation/nitration product of resorcinol and dinitrosoresorcinol were examined. At a precipitation temperature of 40° - 45°C, no change in the physical form resulted. At a precipitation temperature of 80° - 85°C, without additive, the acicular alpha form was obtained; with 2-nitroresorcinol, the cigar-shaped beta crystals were obtained; with dinitrosoresorcinol, granules of the red beta polymorph were formed, and with nitrosation/nitration product of resorcinol minute hairy crystals of the alpha form were precipitated.

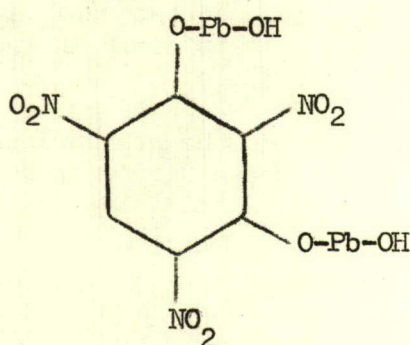
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5. PROPERTIES OF MONOBASIC LEAD STYPHNATE

5.1 Chemical

5.1.1 Analysis

The chemical analysis of the three polymorphic forms of basic lead styphnate gave percentages for lead and styphnate radicals corresponding to the empirical formula $C_6H_3N_3O_{10}Pb_2$, structurally represented by



The analyses found for the various forms were as follows:

alpha form	(common form	Lot XBLS4	Pb 59.90%;	styphnate 34.70%
	(modified form	Lot BLS26	Pb 59.77%;	styphnate 34.20%
beta form	(R.D.1346	Lot XBLS13	Pb 59.80%;	styphnate 34.70%
	(R.D.1349	Lot XBLS15	Pb 59.60%;	styphnate 34.50%
gamma form	Lot BLS21		Pb 59.90%;	styphnate 33.60%

Theory for $C_6H(NO_2)_3O_2Pb \cdot Pb(OH)_2$ requires Pb 59.92%; styphnate 35.16%.

/5.1.2

5.1.2 Action of Acid

The three polymorphic forms are readily hydrolysed by the action of excess dilute nitric or acetic acid with immediate formation of free styphnic acid. The rate of hydrolysis of the monobasic salts is markedly faster than that of the normal alpha or beta lead styphnate monohydrate: this is possibly due to surface heat of neutralisation of the basic radical. The action of equimolecular proportions of aqueous styphnic acid on the three monobasic forms gave in each case the beta polymorph of normal lead styphnate monohydrate. Thus 3.46 g. (0.0025 mole) alpha monobasic form (XBSL 4) suspended in 100 ml. water to which was added 1.5 g. of styphnic acid and left standing for three days at room temperature with occasional stirring gave 4.25 g. of normal beta lead styphnate monohydrate. 3.46 g. beta form R.D.1346 (XBSL56) gave 4.05 g.; 3.46 g. beta form R.D.1349 (XBSL40) gave 4.25 g., and 3.46 g. gamma form (XBSL49) gave 4.30 g. Theoretically 4.28 g. (0.005 mole) of the normal salt should result.

5.1.3 Stability

Both R.D.1346 and R.D.1349 exhibit high thermal stability and are suitable for application where stability above 100°C is important. When heated at 100°C for 7 hours, or at 130°C over phosphorus pentoxide at 10 mm Hg pressure for 4 hours, or heated on a thermogravimetric balance at a rate of 0.7 degC/min. to 200°C no change in physical form or loss in weight occurred. No change occurred when either material was stored under water at ambient temperature for 12 months, the water remaining neutral; they are also non-hygroscopic. They are only very slightly soluble in water (0.0008 g. per 100 ml. solution at 25°C (10) and are compatible with the usual detonator or igniter shell metal and also with the common oxidants such as barium nitrate, potassium nitrate and chlorate.

5.2 Physical

The physical properties of the various forms of monobasic lead styphnates are listed:

TABLE 1

Sample		Absolute Density at 20°C, g/ml.	Apparent Density of Dry Solid, g/ml.
Alpha	(common form (XBSL4) (modified form (XBSL57))	4.14	0.25 1.85
Beta	(R.D.1346 (XBSL56) (R.D.1349 (XBSL40))	4.06	range from 1.0 to 2.0
Gamma	(XBSL49)	4.32	0.52

/5.3

5.3 Explosive5.3.1 Sensitiveness to Heat

Ignition temperature when heated at 5 degC/min:

Alpha common form (XBLS4) 265.6°C; alpha modified form (XBLS57) 260.3°C;
beta R.D.1346 (XBLS56) 248°C - 250°C; beta R.D.1349 (XBLS40) 248°C - 250°C;
gamma form (XBLS49) 248°C - 250°C.

5.3.2 Sensitiveness to Impact and Friction

TABLE 2

Ball and Disc

Explosive	Percentage Ignitions in 50 Trials					Rotter Machine Figure of Insensitiveness
	Height of Fall, cm:					
	4	5	6	8	10	
XBLS4 (Alpha)	-	-	6	64	96	10
XBLS57 (Alpha)	-	-	0	26	92	16
XBLS56 (R.D.1346)	0	4	20	68	100	10
XBLS40 (R.D.1349)	0	4	38	70	100	10
XBLS49	-	-	2	36	80	11
R.D.1303/(M)CY 64/57 Normal Lead Styphnate	0	4	14	-	-	25

Comparison of the five varieties with the normal salt of lead styphnate (R.D.1303M) indicates that all are more sensitive on the figure of insensitiveness index, but on the ball and disc results the beta forms are similar in sensitiveness to R.D.1303M at the low response end, but in turn are a little more sensitive than the alpha and gamma forms.

/TABLE 3

TABLE 3

E.R.D.E. Sliding Block/Emery Paper Test

Explosive	Velocity of Strike, ft/sec:				
	6	7	8	9	10
Alpha, XBLS4	0/10	4/10	4/10	9/10	-
Alpha, XBLS57	2/10	2/10	4/10	7/10	-
Beta, R.D.1346	1/10	4/10	8/10	8/10	-
Beta, R.D.1349	1/10	3/10	4/10	8/10	-
Gamma, XBLS49	1/10	3/10	-	4/10	-
R.D.1303(M) CY 64/57	-	-	0/10	3/10	7/10

The sensitiveness to friction of the five forms are of the same order, but they are more sensitive than the normal lead styphnate R.D.1303M.

5.3.3 Sensitiveness to Electrostatic Spark Ignition

The threshold energies for ignition of the five main varieties of mono-basic lead styphnate and for normal lead styphnate are listed in the Table 4 below. More detailed explanation of the test conditions is given by Wyatt (11).

The common yellow alpha form (XBLS4) and the gamma form (XBLS49) are very sensitive to spark ignition, the modified alpha form (XBLS57) after the method of Hitchens and Garfield is similar to the normal lead styphnate R.D.1303M, whereas R.D.1346 and R.D.1349 are intermediate, with R.D.1349 less sensitive than R.D.1346 to discharge between two metal electrodes.

The correlation between method of preparation, with and without additives, and electrostatic sensitiveness is illustrated in Table 5 (p. 14). Both method of preparation and additive affect sensitiveness. Without additive this explosive whether the alpha, beta or gamma form is extremely sensitive to electrostatic ignition; with the additive nitrosation/nitration product of resorcinol a desensitising action occurs irrespective of method. The additive 2-nitroresorcinol has a slight desensitising effect, and in the development of R.D.1349 the location and distribution of the ammonia used for basicity has a marked desensitising effect. The measurements for XBLS5, XBLS6 and BLS119 illustrate this.

/TABLE 4

TABLE 4

Explosive	Metal/Metal Electrodes		Rubber/Metal Electrodes		
	Min. Energy, ergs	Type of Ignition	Minimum Capacitance, $\mu\mu\text{F}$	Min. Energy, ergs	Type of Ignition
Alpha, XBLS4	26	C	<20*	~30*	C
Alpha, XBLS57	400	P' + B'	~15	250 at 40 $\mu\mu\text{F}$	P
Beta, R.D.1346	40	C	~15	100 at 40 $\mu\mu\text{F}$	C
Beta, R.D.1349	400	C + B	~15	80 at 40 $\mu\mu\text{F}$	C'
Gamma, XBLS49	15	C	<20*	~20*	C
R.D.1303M, CY 23/58	210	P + B	~25	250 at 40 $\mu\mu\text{F}$	P

All the M/M tests were carried out with a capacitance of 520 $\mu\mu\text{F}$

Types of Ignition: C = All complete ignitions
 C' = Nearly all complete, remainder being partial ignitions
 P = Partial ignitions (i.e. a third or more of the explosive ignited)
 P' = Small partial ignition (i.e. just sufficient material ignited to disturb the remainder)
 B = Burn mark (i.e. a blackened mark left on the roller, explosive otherwise disturbed)
 B' = Very small burn marks

*R/M Energy still decreasing at 20 $\mu\mu\text{F}$

5.3.4 Applications

R.D.1349 is in suitable physical form for making satisfactory priming dips for fuseheads. In this connection it can be used to give very sensitive fuseheads, and trials are in progress to meet Specification Air 1447 Type G operating with a current of 0.09 amp. By employing thicker wire, less sensitive fuseheads can be made which will exploit the high thermal stability of R.D.1349 for Service conditions of extreme temperature. The standard plant batch of 3½ lb. produces sufficient R.D.1349 for very large numbers of fuseheads.

R.D.1346 has potential application to bridge wire igniters where dry loading is required. It can also be mixed with finely divided graphite to give a conducting composition. These applications are being investigated especially in connection with designs for high temperature resistant stores.

/6.

6. CONCLUSIONS

6.1 The alpha polymorph of monobasic lead styphnate is unsuitable for technical application unless modified to improve physical form and reduce electrostatic spark sensitiveness. The nitration/nitrosation complex of resorcinol is confirmed as an additive offering substantial improvement. This additive is an ill-defined mixture which is difficult to specify and further examination in order to isolate the active ingredient is proceeding.

6.2 The beta polymorph is less objectionable but requires very careful control of preparation to give a consistent satisfactory product on the technical scale. By using a simple additive, mononitroresorcinol, processes have been developed for two new compositions (R.D.1346 and R.D.1349) of promising technical value. Consideration of these compositions is recommended, especially for electric fuseheads and stores liable to be subjected to high temperature in Service.

6.3 The gamma polymorph is not recommended for further study or development.

7. ACKNOWLEDGEMENTS

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/TABLE 5

Electrostatic Sensitiveness

Batch No.	Method of Preparation	Additive	Nature of Mater
XBLS4 } XBLS14 } XBLS2 } XBLS9 } XBLS56 }	Addition of sodium styphnate solution to lead nitrate at 70°C	None None 2-Nitroresorcinol ditto	Alpha yellow aci ditto Beta, R.D.1346 rounded ditto ditto
XBLS5 } XBLS64 } XBLS65 } BLS121 } BLS122 }	Addition of lead acetate to ammonium styphnate at 70° - 75°C	2-Nitroresorcinol ditto ditto N/N product None	Beta, R.D.1349 oval sha ditto ditto Alpha, fine pow Alpha, matted ne
XBLS6 } XBLS40 } XBLS72 } XBLS77 }	Addition of ammoniacal lead acetate to ammonium styphnate at 70° - 75°C	2-Nitroresorcinol ditto ditto ditto	Beta, R.D.1349 oval sha ditto ditto ditto
BLS21 } XBLS19 }	Addition of lead nitrate to ammonium styphnate at 40°C	None None	Gamma, amorphous yell ditto
BLS26 XBLS57 } BLS118 } BLS119 }	Hitchens & Garfield method Addition of ammonia and lead nitrate to ammonium styphnate at 70°C	N/N product ditto ditto None	Alpha, modified paral crystals ditto ditto Alpha, unmodified aciou
BLS114 } BLS123 } BLS124 }	Addition of lead nitrate to sodium styphnate at 90°C	None N/N product 2-Nitroresorcinol	Alpha yellow malted Alpha fine pow ditto

N/N product = Nitrosation/nitration product of resorcinol.

TABLE 5

2

Sensitivity of Basic Lead Styphnate

Material	Metal/Metal Electrodes		Rubber/Metal Electrodes		
	Minimum Energy, ergs	Type of Ignition	Minimum Capacitance $\mu\mu\text{F}$	Minimum Energy, ergs	Type of Ignition
low acicular	26	C	<20*	~30*	C
itto	10	C	-	-	-
ounded red granules	85	C	-	-	-
itto	80	C	-	-	-
itto	40	C	~15	100 at 40 $\mu\mu\text{F}$	C
val shaped crystals	25	C	-	-	-
itto	26	C	<20	20 at 25 $\mu\mu\text{F}$	C
itto	30	C	-	-	-
ne powder	300	C	-	-	-
ted needles	~5	C	-	-	-
val shaped crystals	320	P' + B	-	-	-
itto	400	C + B	~15	100 at 40 $\mu\mu\text{F}$	C'
itto	400	C + P + B	<20*	~40*	C
itto	520	C + B	-	-	-
as yellow powder	25	C	-	-	-
itto	15	C	<20*	~20*	C
l parallelopiped crystals	250	C + P	-	-	-
itto	400	P' + B'	~15	250 at 40 $\mu\mu\text{F}$	P
itto	120	C	-	-	-
l acicular crystals	~5	C	-	-	-
alted crystals	~4	C	-	-	-
ne powder	15	C	-	-	-
itto	~5	C	-	-	-

Notes All the M/M tests were carried out with a capacitance of 520 $\mu\mu\text{F}$, except with XBS14 (220 $\mu\mu\text{F}$) and BLS21 (320 $\mu\mu\text{F}$)

Type of Ignition: C = All complete ignition

C' = Nearly all complete, remainder being partial ignitions

P = Partial ignitions (i.e. a third or more of the explosive ignited)

P' = Small partial ignition (i.e. just sufficient material ignited to disturb the remainder)

B = Burn mark (i.e. a blackened mark left on the roller, explosive

parallel piped als	250	C + P	-	-	-
o	400	P' + B'	~15	250 at 40 $\mu\mu\text{F}$	P
o	120	C	-	-	-
cicular crystals	~5	C	-	-	-
ted crystals	~4	C	-	-	-
powder	15	C	-	-	-
o	~5	C	-	-	-

3

Notes All the M/M tests were carried out with a capacitance of 520 $\mu\mu\text{F}$, except with XBLS14 (220 $\mu\mu\text{F}$) and BLS21 (320 $\mu\mu\text{F}$)

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B = Burn mark (i.e. a blackened mark left on the roller, explosive otherwise undisturbed)

B' = Very small burn marks

*R/M Energy still decreasing at 20 $\mu\mu\text{F}$

/APPENDIX

APPENDIX:

METHODS OF MANUFACTURE OF R.D.1346 and R.D.1349

I THE PREPARATION OF R.D.1346

A Semi-technical Scale

Plant 10-litre stainless steel jacketed pan, 4-inch stirrer at approx. 250 rev/min. Temperature maintained by circulating hot water from a calorifier. Mercury-in-steel 0 - 100°C thermometer fitted in pan.

Ingredients

1. Sodium styphnate solution containing 36.768 g. styphnic acid; 0.80 g. 2-nitroresorcinol; 16.12g. sodium carbonate per litre.
2. Sodium hydroxide solution containing 80 g. NaOH per litre.
3. Lead nitrate solution containing 250 g. $\text{Pb}(\text{NO}_3)_2$ per litre.

Process Instructions

1. Add 1.987 litres of lead nitrate solution to the pan and adjust temperature to 70°C with stirring.
2. Mix together 2.5 litres of sodium styphnate solution and 0.345 litre sodium hydroxide solution, make up to 3.0 litres with water and add in a steady stream to the pan during 60 minutes.
3. Continue stirring at 70°C for a further 5 minutes.
4. Stop stirrer and allow product to settle for 2 minutes.
5. Decant mother-liquor.
6. Wash product twice, by decantation, with 5 litres of distilled water for each wash.
7. Filter. Wash product with 2 litres of industrial methylated spirit.
8. Dry on hot table at 50°C or by cold air.
9. Sieve through 40 mesh B.S.S. stainless steel screen.

Special Instructions

1. Styphnic acid of adequate purity (Specification C.S.1936 applies) must be used.
2. Full electrostatic precautions must be taken when product is dry.

/B

B Full Plant Scale

Plant 10-gallon stainless steel jacketed pan with stainless steel stirrer at 76 - 83 rev/min. Mercury-in-steel thermometer fitted in pan, temperature maintained by passing live steam through pan jacket.

Ingredients

As for A

Process Instructions

1. Add 11.92 litres of lead nitrate solution to the pan and adjust temperature to 70°C with stirring.
2. Mix together 15.0 litres of sodium styphnate solution and 2.07 litres sodium hydroxide solution, make up to 18.0 litres with water and add in a steady stream to the pan in 90 minutes.
3. Continue stirring at 70°C for a further 5 minutes.
4. Stop stirrer and allow product to settle for 2 minutes.
5. Decant mother-liquor.
6. Wash product twice by decantation with 25 litres of distilled water for each wash.
7. Filter into two gutta-percha drying pots, wash with 2 litres of methylated spirit per pot.
8. Dry by passing cold air - 250 ft.³ per pot.
9. Sieve through 40 B.S.S. stainless steel screen.

Special Instructions

As for A. Yield 3½ lb. Bulk density 1.2 g/ml.

II THE PREPARATION OF R.D.1349

C Semi-technical Scale

Plant

As for A

Ingredients

1. Ammonium styphnate solution made up to 3.0 litres containing 147.1 g. styphnic acid, 36.5 g. ammonia (NH₃) and 3.0 g. 2-nitroresorcinol.
2. Lead acetate solution made up to 1.60 litres containing 478 g. Pb(CH₃COO)₂·3H₂O and 4.25 g. ammonia.

/Process

Process Instructions

1. Add the ammonium styphnate solution to the pan and heat to 75°C with stirring.
2. Add lead acetate/ammonia solution in a steady stream during 40 minutes.
3. Continue stirring at 75°C for a further 5 minutes.
4. Stop stirrer and allow product to settle for 5 minutes.
5. Decant mother-liquor.
6. Wash product twice, by decantation, with 4 litres of distilled water for each wash.
7. Filter. Wash product with 2 litres of industrial methylated spirit.
8. Dry product by cold air, or on a hot table at 50°C.
9. Can be stored wet under water.

Special Precautions

As for A

D Full Plant Scale

Plant

As for B

Ingredients

1. Ammonium styphnate solution made up to 18.0 litres containing 882.6 g. styphnic acid, 18.0 g. 2-nitroresorcinol and 219.3 g. ammonia.
2. Lead acetate solution made up to 10 litres containing 2870 g. $\text{Pb}(\text{CH}_3\text{COO})_2 \cdot 3\text{H}_2\text{O}$ and 25.5 g. ammonia.

Process Instructions

1. Add the ammonium styphnate solution to the pan and heat to 75°C with stirring.
2. Add lead acetate solution in a steady stream during 50 minutes.
3. Continue stirring at 75°C for a further 5 minutes.
4. Stop stirrer and allow product to settle for 5 minutes.
5. Decant mother-liquor.
6. Wash product twice, by decantation, with 20 litres of distilled water for each wash.
7. Filter into two gutta-percha drying pots, and wash with 2 litres of industrial methylated spirit per pot.
8. Dry by passing cold dry air or store under water.

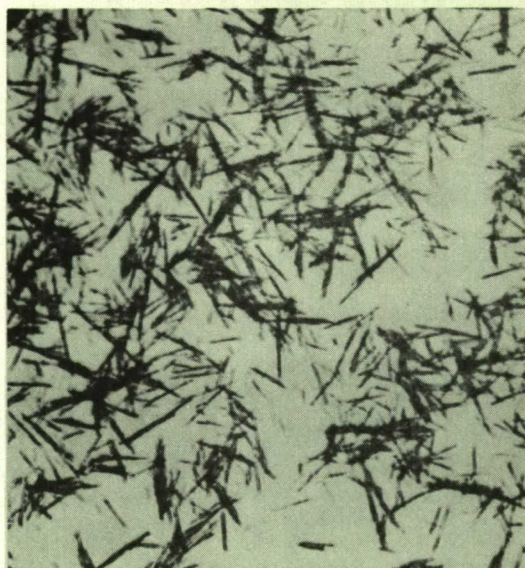
Special Precautions

As for A Yield 4¼ lb.

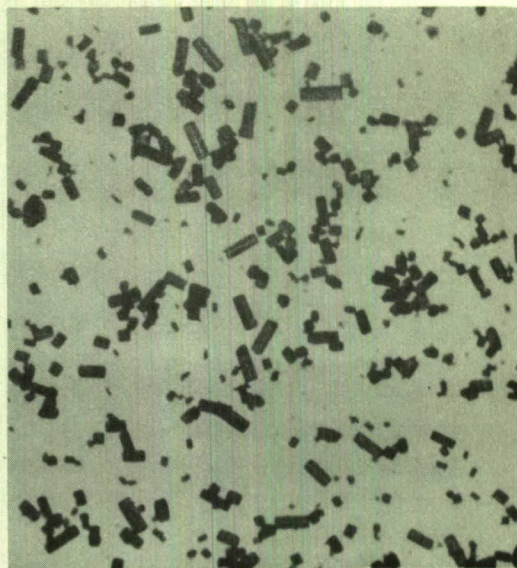
S. No. 785/BL/62

MONOBASIC LEAD STYPHATE.

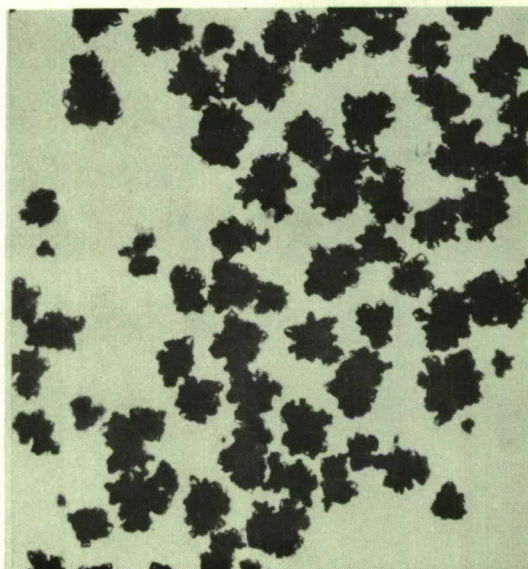
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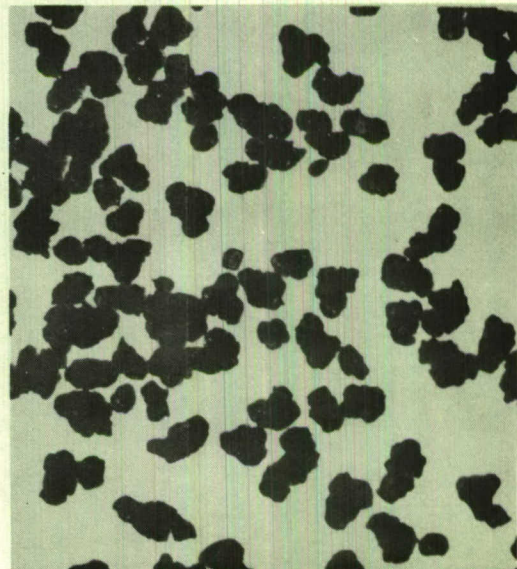
ALPHA COMMON FORM XBLS 4
FIG. 1.



ALPHA MODIFIED FORM XBLS 57
FIG. 2.



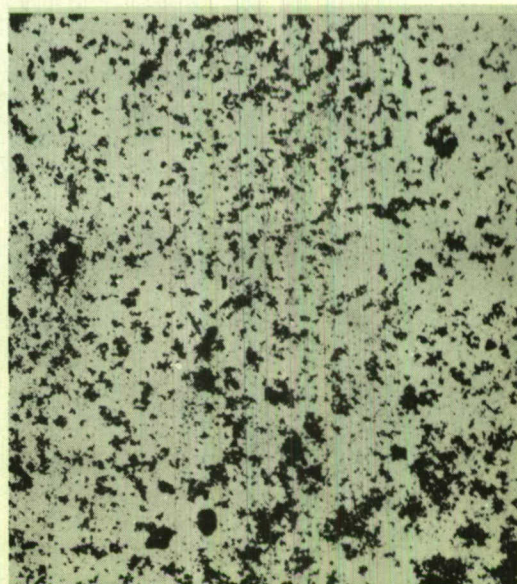
BETAFORM RD 1346 XBLS 9
BULK DENSITY 0.80 g/ml. FIG. 3.



BETAFORM RD 1346 XBLS 13
BULK DENSITY 1.70 g/ml FIG. 4.



BETAFORM RD 1349 XBLS 77
FIG. 5.



GAMMA FORM XBLS 49
FIG. 6.

CRDSOU-Q

1 May 1969

SUBJECT: Current Classification of ERDE Reports

27 January 1969

Classification of British ERDE Reports
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G.W.C. Taylor and A.T. Thomas

June, 1962

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17 pp., 6 fig., 5 tables.

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